CLAIMS

1. Process for the treatment of the reaction mixture resulting from the direct oxidation of cyclohexane to adipic acid by molecular oxygen in an organic solvent in the presence of a catalyst, characterized in that the said process comprises:

- a separation into two liquid phases by settling: an upper phase, which is essentially cyclohexane, and a lower phase, essentially comprising the solvent, the diacids formed, the catalyst and a portion of the other reaction products and of the unconverted cyclohexane;

- a distillation of the said lower phase, making it possible to separate, on the one hand, a distillate comprising at least a portion of the most volatile compounds, such as the organic solvent and water, as well as unconverted cyclohexane, cyclohexanone, cyclohexanol, cyclohexyl esters and lactones possibly present, and, on the other hand, the distillation bottoms comprising the diacids formed and the catalyst;

- a separation of the catalyst from the distillation bottoms obtained above, either by crystallization from water, by electrodialysis or by passing over an ion-exchange resin, after dissolution of the said distillation bottoms in water, or alternatively by washing with water or by liquid-liquid

extraction;

- a reducing and/or oxidizing purification treatment of the adipic acid in aqueous solution;

- a crystallization, preceding or following the purification treatment, when the crystallization has not been carried out in order to separate the catalyst;

- a recrystallization of the adipic acid from water.

2. Process according to claim 1, characterized in that the cyclohexane phase obtained in the stage of separation by settling is reintroduced into a cyclohexane oxidation operation.

3. Process according to either of claims 1 and 2, characterized in that the organic solvent employed in the oxidation of the cyclohexane is chosen from aliphatic carboxylic acids and is preferably acetic acid.

4. Process according to one of claims 1 to 3, characterized in that the catalyst comprises cobalt, manganese or a mixture of cobalt with one or more other metals chosen from manganese, chromium, iron, zirconium, hafnium or copper.

5. Process according to one of claims 1 to 4, characterized in that the stage of distillation of the lower phase is carried out so that most, preferably virtually all, of the unconverted cyclohexane still present in this lower phase and of the solvent is separated from the adipic acid.

6. Process according to one of claims 1 to 5, characterized in that the distillation stage is carried out at a temperature of 25°C to 250°C and under an absolute pressure of between 10 Pa and atmospheric pressure and preferably at a temperature situated between 70°C and 150°C.

7. Process according to one of claims 1 to 6, characterized in that the distillation stage is completed by an extraction of the distillation bottoms using a water-immiscible organic solvent.

8. Process according to claim 7, characterized in that the extraction is carried out with an organic solvent chosen from aliphatic, cycloaliphatic or aromatic hydrocarbons, aliphatic, cycloaliphatic or aromatic carboxylic acid esters, and ketones, and preferably with cyclohexane.

9. Process according to one of claims 1 to 8, characterized in that the distillation bottoms obtained at the end of the distillation, which have been subjected, if appropriate, to the extraction operation, are treated, in order to separate the catalyst which they comprise, by a crystallization operation or by an electrodialysis or by passing the said solution over an ion-exchange resin or by one or more washing operations with water.

10. Process according to one of claims 1 to 9, characterized in that the purification is carried out by hydrogenation and/or by treatment with nitric

acid and/or by oxidation using molecular oxygen, ozone or hydroperoxide.

11. Process according to claim 10, characterized in that the purification by hydrogenation is carried out using hydrogen in the presence of a catalyst.

12. Process according to either of claims 10 and 11, characterized in that the catalyst comprises at least one metal from group VIII of the Periodic Classification of the Elements, such as palladium, platinum, ruthenium, osmium, rhodium, iridium, nickel and cobalt, preferably deposited on a solid support.

13. Process according to claim 10, characterized in that the purification by treatment with nitric acid is carried out with an aqueous solution comprising from 20% to 80% of pure nitric acid by weight per weight of solution.

14. Process according to claim 13, characterized in that the treatment with nitric acid is carried out by heating the mixture at a temperature of 25°C to 120°C and preferably of 40°C to 100°C for a period of time of a few minutes to a few hours.

15. Process according to either of claims 13 and 14, characterized in that the treatment with nitric acid is carried out in the absence of catalyst or in the presence of a catalyst comprising one or more cobalt, copper and/or vanadium compounds.

16. Process according to claim 10,

characterized in that the purification by oxidation is carried out with air, air enriched in oxygen or air depleted in oxygen, in the presence of a catalyst.

17. Process according to claim 16, characterized in that the catalyst is a metal from group VIII of the Periodic Classification of the Elements chosen from palladium, platinum, ruthenium, osmium, rhodium or iridium.

18. Process according to claim 10, characterized in that the purification by oxidation using a hydroperoxide is carried out with hydrogen peroxide.

19. Process according to one of claims 1 to 18, characterized in that the reducing and/or oxidizing purification treatment is followed by an operation of crystallization and/or of recrystallization of the adipic acid from water.

20. Process according to one of claims 1 to 19, characterized in that the reducing and/or oxidizing purification treatment is preceded or is followed by a treatment for adsorption of impurities by a finely divided solid.

PROCESS FOR SEPARATION AND PURIFICATION OF ADIPIC ACID

The present invention relates to the treatment of the reaction mixtures resulting from an oxidation reaction of cyclohexane to adipic acid and more particularly to the separation of the various constituents of the said mixtures and to the purification of the adipic acid.